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TECHNICAL Report No. 9

AEROSOL DIRECT FLUORINATION "INDIRECT SYNTHESES OF PERFLUOROCYCLOKETONES"

by

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Ring opening during the aerosol direct fluorination of cyclic ketones can be circumvented by the aerosol direct fluorination of the appropriate methoxycycloalkanes or the ethylene glycol ketals of the cyclic ketones followed by sulfuric acid hydrolyses of the perfluorinated analogs. Aerosol direct fluorinations of methoxycyclopentane and methoxycyclohexane produce their respective perfluoroanalogs in effluent concentrations of 57% and 90% by weight and in 22% and 32% isolated yields respectively. Sulfuric Acid

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hydrolyses at 340° to 360°C of the F-methoxycycloalkanes produced F-cyclo-pentanone (89% yield, 61% conversion) and F-cyclohexanone (82% yield, 28% conversion) respectively. The aerosol fluorinations of the ethylene glycol ketals of cyclopentanone and cyclohexanone produce F-dioxaspiro [4.4] nonane and F-dioxaspiro [4.5] decane in effluent concentrations of 74% and 76% by weight and in 14% and 12% isolated yields respectively. Sulfuric acid hydrolyses at 500°C produced F-cyclopentanone (45% yield, 23% conversion) and F-cyclohexanone (100% yield, 36% conversion) respectively. Physical loss of starting material caused by condensation within the aerosol fluori-nator is the major reason for reduced yields. Hydrolysis yields are based on starting materials consumed and not recovered.

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Aerosol Direct Fluorination – Indirect Syntheses of Perfluorocycloketones

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Ring opening during the aerosol direct fluorination of cyclic ketones can be circumvented by the aerosol direct fluorination of the appropriate methoxycycloalkanes or the ethylene glycol ketals of the cyclic ketones followed by sulfuric acid hydrolyses of the perfluorinated analogs. Aerosol direct fluorinations of methoxycyclopentane and methoxycyclohexane produce their respective perfluoroanalogs in effluent concentrations of 57% and 90% by weight and in 22% and 32% isolated yields respectively. Sulfuric Acid hydrolyses at 340° to 360°C of the F-methoxycycloalkanes produced F-cyclopentanone (89% yield, 61% conversion) and F-cyclohexanone (82% yield, 28% conversion) respectively. The aerosol fluorinations of the ethylene glycol ketals of cyclopentanone and cyclohexanone produce F-dioxaspiro[4.4]nonane and F-dioxaspiro[4.5]decane in effluent concentrations of 74% and 76% by weight and in 14% and 12% isolated yields respectively. Sulfuric acid hydrolyses at 500°C produced F-cyclopentanone (45% yield, 23% conversion) and F-cyclohexanone (100% yield, 36% conversion) respectively. Physical loss of starting material caused by condensation within the aerosol fluorinator is the major reason for reduced yields. Hydrolysis yields are based on starting materials consumed and not recovered.

Aerosol Direct Fluorination - Indirect Syntheses of Perfluorocycloketones

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The aerosol direct fluorination method provides a continuous process for the production of perfluorocarbons from hydrocarbons with efficient fluorine utilization and minimal fragmentation.[1] The application of this process to alkanes, ethers, cycloalkanes, ketals and ketones has been demonstrated.[1-3] Whereas the aerosol direct fluorination of alicyclic ketones yields the corresponding perfluoroketone as the majority product,[2] aerosol fluorination of cyclic ketones has produced none of the perfluorinated analogs. In the case of cyclopentanone, the major product was F-pentanoyl fluoride, resulting from the opening of the cyclopentyl ring[2]; aerosol direct fluorination of cyclohexanone produced only non-volatile products; no evidence for the formation of F-cyclohexanone was found.[4] However, employing a two-step process perfluorocyclopentanone and perfluorocyclohexanone have been synthesized. Sulfuric acid hydrolysis of the appropriate perfluorinated ketal or ether, formed via aerosol fluorination of the corresponding ketal or ether, produced the corresponding perfluorocycloketone in high yields.

Previous preparations of F-cyclopentanone and F-cyclohexanone are relatively few in number; the only synthesis involving direct fluorination is that reported by Holub and Bigelow wherein F-cyclopentanone was formed in trace amounts from the direct fluorination of cyclopentanone.[5] F-cyclopentanone and F-cyclohexanone have been prepared via the treatment of the corresponding 2,2-dichloroperfluoro cyclic ketones or 1,2-dichloroperfluorocycloalkene epoxides with potassium fluoride in tetramethylene sulfone[6], and

1

by the contact of Lewis acids or bases with the appropriate perfluoro olefin epoxides.[7] In addition to the above methods, Tatlow, et al.,[8] prepared F-cyclopentanone by the cleavage of methyl nonafluorocyclopentyl ether with sulfuric acid, and F-cyclohexanone by the cleavage of methyl or fluoromethyl undecafluorocyclohexyl ether with sulfuric acid.

Discussion

The syntheses of F-cyclopentanone and F-cyclohexanone were effected via two separate two-step reaction sequences. Scheme 1 outlines the syntheses of the perfluorocycloketones in a two-step process from the corresponding methyl ethers. F-Methoxycyclopentane (IIa) and F-methoxycyclohexane (IIb) obtained from the aerosol direct fluorination of the corresponding hydrocarbon ethers (Ia,Ib) in 22% and 32% yields respectively, were converted to F-cyclopentanone (IIIa) and F-cyclohexanone (IIIb) by hydrolysis with 100% sulfuric acid at 340°-360°C in 89% and 82% yields respectively. F-methoxycyclopentane and F-methoxycyclohexane comprised 57% and 90% respectively of the total products collected by weight from the aerosol reactor. Physical losses due to unfluorinated starting materials "freezing out" in the first reaction zone reduce overall yields to the values cited.[9] F-methoxycyclopentane and F-methoxycyclohexane have been previously prepared by the

Scheme 1

$$(CH_2)_n \begin{matrix} CH_2-CH-O-CH_3 \\ | \\ CH_2-CH_2 \end{matrix} \xrightarrow{AF} (CF_2)_n \begin{matrix} CF_2-CF-O-CF_3 \\ | \\ CF_2-CF_2 \end{matrix} \xrightarrow[340°-360°C]{H_2SO_4} (CF_2)_n \begin{matrix} CF_2-C=O \\ | \\ CF_2-CF_2 \end{matrix}$$

Ia, n = 1   IIa, n = 1   IIIa, n = 1

Ib, n = 2   IIb, n = 2   IIIb, n = 2
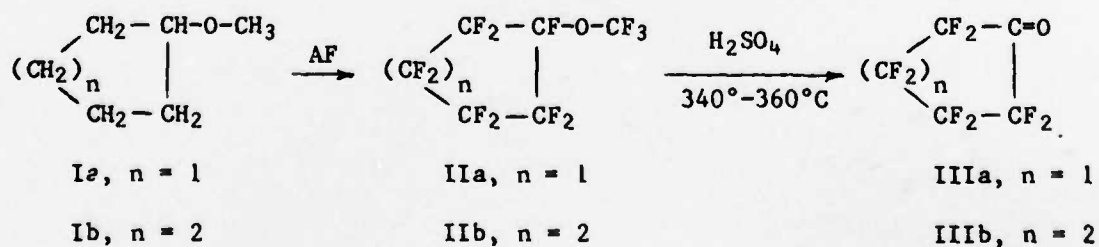
photochemical reaction of trifluoromethyl hypofluorite and the corresponding perfluorocycloalkene.[10,11]

The perfluoroethers IIa and IIb are stable compounds: after 16 hours at 200°C in the presence of 100% sulfuric acid, F-methoxycyclopentane showed no signs of reaction. At 340-360°C in the presence of 100 % sulfuric acid both perfluoroethers form their respective perfluoroketones (IIIa,IIIb) in high yields. In each case only the perfluoroketone and unreacted perfluoroether were found in the reaction mixture. Any other products were present only in trace amounts.

Both perfluoroketones are extremely hygroscopic and react readily with water to form the much less volatile monohydrates[6a]. This suggests that in the aerosol direct fluorinations of cyclopentanone[2] and cyclohexanone[4] any perfluoroketone formed might have become hydrated due to reaction with residual water inside the reactor, explaining the fact that no perfluoroketones could be isolated from those reactions.

Scheme 2 outlines the syntheses of F-cyclopentanone and F-cyclohexanone in a two-step process from the appropriate ketals. Aerosol direct

Scheme 2

| IVa, n = 1 | Va, n = 1 | IIIa, n = 1 |
| IVb, n = 2 | Vb, n = 2 | IIIb, n = 2 |
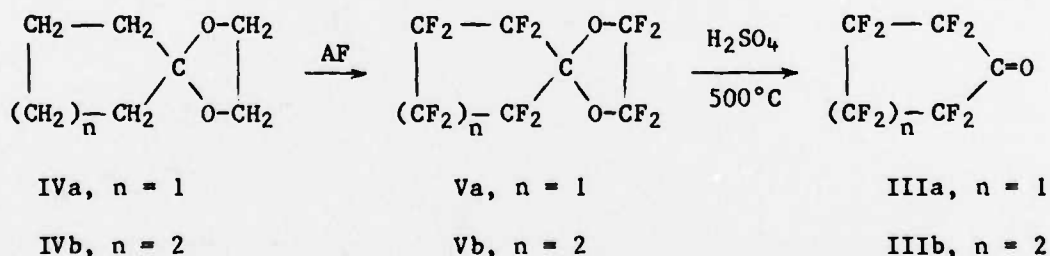
fluorinations of 1,4-dioxaspiro[4.4]nonane (IVa) and 1,4-dioxaspiro[4.5]-decane (IVb) produce the previously unknown F-1,4-dioxaspiro[4.4]nonane (Va) and F-1,4-dioxaspiro[4.5]decane (Vb) (74% and 76% respectively of the total product collected by weight) in percent yields based on throughput of 14% and 12%, respectively. As previously mentioned, low yields are mostly due to physical losses in the reactor.[9]

Both perfluoroketals are extremely stable compounds: F-1,4-dioxaspiro-
[4.5]decane showed no signs of reaction after 26 hours at 350°C in the
presence of 100 % sulfuric acid. F-1,4-Dioxaspiro[4,4]nonane (Va) and
F-1,4-dioxaspiro[4,5]decane (Vb) were, however, converted to F-cyclopen-
tanone and F-cyclohexanone via hydrolysis with 100% sulfuric acid at 500°C
in 45% and in 100% yield respectively. In the case of F-cyclopentanone, the
lower yield is believed to be due to more extensive hydration of the
perfluoroketone at the higher temperature. F-cyclopentanone is more
affected because more ring-strain would be relieved upon hydrate formation
than in the case of F-cyclohexanone, and hence one would predict F-cyclopen-
tanone to be more hygroscopic than F-cyclohexanone.

Experimental

The basic aerosol fluorinator design and a basic description of the
process are presented elsewhere[1]; a modified aerosol generator was adapted
to a flash evaporator fed by a syringe pump (Sage Model 341a) driving a
5.000 ml Precision Sampling Corp. "Pressure-lok" syringe.[3] Workup of
products following removal of hydrogen fluoride consisted of vacuum line
fractionation, infrared assay of fractions, gas chromatographic separation
of components using either a 7 meter x 3/8" 13 % Fluorosilicone QF-1
(Analabs) stationary phase on 60-80 mesh, acid washed Chromosorb P condi-
tioned at 225°C (12 hours), or a 4 meter x 3/8" 10 % SE-52 phenyl-methyl
silicone rubber on acid washed 60-80 mesh Chromosorb P conditioned at 250°C
(12 hours). Following gas chromatographic separation (Bendix Model 2300,
sub-ambient multi-controller) all products of significance were collected,
transferred to the vacuum line, assayed and characterized by vapor phase
infrared spectrophotometry (Perkin Elmer 1330), electron impact (70 eV) and
chemical ionization (CH_4 plasma) mass spectrometry (Hewlett Packard GC/MS,

5710A GC, 5980A MS, 5934A computer), and ^{19}F nuclear magnetic resonance (JEOL FX90Q, omniprobe) in CDCl₃ with 1 % CFCl₃ internal standard. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, N.Y. The above characterizations and detailed aerosol reaction parameters (6 pages) are available as Supplementary Material, ordering information is given on any current masthead page.

Aerosol Fluorination of Methoxycyclopentane. Methoxycyclopentane was prepared by the method of Vogel from cyclopentanol.[11] A pump speed corresponding to 3.7 mmol per hour was established and 2.40 ml (2.05g, 20.5 mmol) methoxycyclopentane was delivered over a 5.5 hour period. Details of the aerosol parameters are available as Supplementary Materials. For the photochemically finished reaction 2.42g of crude product was isolated; separation on the QF-1 column (temperature program: 20°C, 10 min; 5°C/min to 100°C; 100°C, 1 min; 50°C/min to 180°C) yielded 1.39g (57 %) pure F-methoxycyclopentane, corresponding to a yield of 22 % based on total methoxycyclopentane injected. The ^{19}F nmr spectrum of F-methoxycyclopentane agreed with that appearing in the literature,[11]

Hydrolysis of F-methoxycyclopentane. 0.321g of F-methoxycyclopentane was condensed onto an approximately equal volume of fuming sulfuric acid (30 % SO₃) which had been degassed and vacuum-stripped to remove the SO₃; the reaction tube was then sealed under vacuum and heated in a tube furnace at 360° for 24 hours. The tube was then opened on the vacuum line and the reaction mixture fractionated through -131°C and -196°C cold traps, most of the material collected in the -131°C trap. Products from the -131°C fraction were separated on the SE-52 column (temperature program: -10°C, 5 min; 0.5°C/min to 20°C) and identified as F-cyclopentanone[11] (.140g) and unreacted F-methoxycyclopentane (.103g). Percent yield of F-cyclopentanone

based on F-methoxycyclopentane reacted was thus 89%, the percent conversion for the reaction was 61%.

Aerosol Fluorination of methoxycyclohexane. Methoxycyclohexane was prepared by the method of Vogel from cyclohexanol.[12] A pump speed corresponding to 3.8 mmol per hour was established and 3.30 ml (2.89g, 25.3 mmol) of methoxycyclohexane delivered over a 6.75 hour period. From the 3.32g of crude product collected 2.99g (90%) of pure F-methoxycyclohexane was isolated (QF-1 program: 35°C, 10 m; 4°C/m to 100°C, 100°C/m; 50°C/m to 180°C) which corresponded to a yield of 32% based on total methoxycyclohexane injected. The ^{19}F nmr spectrum of F-methoxycyclohexane was in agreement with that appearing in the literature.[11]

Hydrolysis of F-methoxycyclohexane. 0.308g of F-methoxycyclohexane was treated with 100% sulfuric acid as described above and heated for 14 hours at 340°C. After workup of the reaction mixture as for F-methoxycyclopentane, separation on the SE 52 column (0°C, 10 m; 1°C/m to 100°C) yielded F-cyclohexanone[11] (0.066g) and unreacted F-methoxycyclohexane (.202g). The percent yield of F-cyclohexanone based on the amount of F-methoxycyclohexane reacted was 82%, the percent conversion to product for the reaction was 28%.

Aerosol Fluorination of 1,4-Dioxaspiro[4.4]nonane. 1,4-Dioxaspiro-[4,4]nonane was prepared by the method of Daignault and Eliel from cyclopentanone.[13] A pump speed corresponding to 4.2 mmol per hour was established, and 3.0 ml (3.21g, 25.1 mmol) of 1,4-dioxaspiro[4.4]nonane was delivered over a 6 hour period. Details of the aerosol fluorination parameters are available as Supplementary Materials. The crude product (1.65g) was separated on the QF-1 column (30°C, 5 m; 5°C/m to 100°C; 100°C,

1 m; 50°C/m to 180°C) and yielded 1.22g (74%) pure F-1,4-dioxaspiro[4.4]-
nonane, corresponding to a 14% yield based on total 1,4-dioxaspiro[4.4]-
nonane injected. The ^{19} NMR consists of *three* singlets of equal intensity at
ϕ = -85.32 ppm; -130.36 ppm and -131.69 ppm. Elemental Analysis: Calcu-
lated for $C_7F_{12}O_2$: C 24.44%, F 66.26%; Found C 24.22%, F 66.25%.

Hydrolysis of F-1,4-Dioxaspiro[4.4]nonane. 0.156g F-1,4-Dioxaspiro-
[4.4]nonane was treated with 100% sulfuric acid (as prepared previously) and
was heated for 24 hours at 450°C. Separation of the products on the
SE-52 column (-10°C, 5 min; 0.5°C/min to 50°C) yielded 0.024g F-cyclopen-
tanone (isolated as the monohydrate) and 0.076g unreacted F-1,4-dioxaspiro-
[4.4]nonane. The percent yield of F-cyclopentanone based on the amount of
F-1,4-dioxaspiro[4.4]nonane reacted was 45%, the percent conversion to
product for the reaction was 23%.

Aerosol Fluorination of 1,4-Dioxaspiro[4.5]decane. 1,4-Dioxaspiro-
[4.5]decane was prepared by the method of Daignault and Eliel from cyclo-
hexanone.[13] A pump speed corresponding to 4.3 mmol per hour was established
and 3.0 ml (3.1g, 21.5 mmol) of 1,4-dioxaspiro[4.5]decane was delivered over
a 5 hour period. The crude products (1.28g) were separated on the QF-1
column (50°C, 5 m; 2°C/m to 100°C; 100°C, 1 m; 50°C/m to 180°C) and yielded
0.97g (76 %) pure F-1,4-dioxaspiro[4.5]decane, corresponding to a percent
yield of 12 % based on total 1,4-dioxaspiro[4.5]decane injected. F-1,4-
Dioxaspiro[4.5]decane ^{19}F NMR consisted of a singlet at ϕ = -83.22 ppm and a
broad multiplet at ϕ = -132.02 ppm of 4:10 relative intensity. Elemental
Analyses: Calculated for $C_8F_{14}O_2$ C 24.38%, F 67.49%; found C 23.85%, F
66.78%.

Hydrolysis of F-1,4-dioxaspiro[4.5]decane. 0.198g F-1,4-Dioxaspiro-
[4.5]decane was treated with 100% sulfuric acid (as prepared previously)

and was heated for 18 hours at 500°C. Separation of the products on the
SE-52 column (0°C, 10 m; 1°C/m to 100°C) yielded 0.050g F-cyclohexanone and
0.127g unreacted F-1,4-dioxaspiro[4.5]decane, both identified from their
infrared spectra. The percent yield of F-cyclohexanone based on F-1,4-
dioxaspiro[4.5]decane reacted was 100 %, the percent conversion to products
for the reaction was 36%.

Supplementary Material Available: Characterization data for products (IR,
MS, ^{19}F NMR, Aerosol Parameters; 6 pages). Ordering information is given on
any current masthead page.

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      aerosol which is ideally crystalline, of uniform size and with little
      tendency to aggregate. If the conditions for producing the aerosol are
      ideal, percent yields based on throughputs and product percent distri-
      butions will differ by only a few percent; as molecules deviate from
      this ideality, the percent yields based on throughput fall, due to
      physical losses within the aerosol generator and initial reaction stage
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APPENDIX I

Supplementary Material

Characterization of Intermediate Products

SUPPLEMENTARY MATERIAL

TABLE 1

TYPICAL AEROSOL FLUORINATION REACTION PARAMETERS

Starting Compound	Fluorine Flow (ml/m)			Helium Flow (ml/m)						Reaction T(°C)				Hydrocarbon throughput mmole/hr	Produced distribution % collected	Product Yield % Theoretical
	Mod 1	Mod 2	Mod 3	main	1°	2°	Mod 1	Mod 2	Mod 3	Evap	Mod 1	Mod 2	Mod 3			
Methoxycyclopentane	15	20	30	500	400	50	150	150	150	150	-20	-10	RT	3.7	57.4	21.5
Methoxycyclohexane	20	20	40	800	750	50	150	150	150	160	-30	-20	RT	3.8	90.0	32.3
1,4-Dioxaspiro[4.4]nonane	10	20	30	700	650	50	150	150	150	175	-20	-10	RT	4.2	74.0	14.2
1,4-Dioxaspiro[4.5]decane	20	20	40	750	650	50	150	150	150	225	-20	-10	RT	4.3	76.1	11.5

SUPPLEMENTARY MATERIAL

TABLE 2

CHARACTERIZATION OF PRODUCTS

F-Methoxycyclopentane
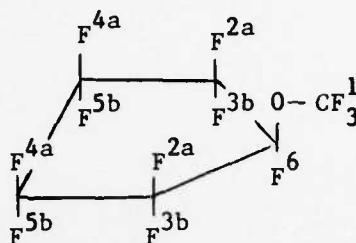
IR: 1320 m, 1280 vs, 1235 vs, 1215 s, 1190 s, 1150 m, 995 s, 985 s, 890 w, 855 w, 730 w, 660 w cm^{-1}

^{19}F nmr:[a]

Chemical Shift, ϕ (ppm)	Int.	coupling constants, Hz	assignment
-55.42	3	$J_{16} = 10.0$	1
		$J_{12} = 6.0$	
		$J_{13} = 3.9$	
-124.68			2a
	4	$J_{2a,3b} = 259.4$	
-135.68			3b
-129.34			4a
	4	$J_{4a,5b} = 254.8$	
-132.86			5b
-137.96	1	Complex multiplet	6

Mass Spectra[a,b]

EI(70eV): 297 (1) $C_6F_{11}O$, M-F; 231 (8) C_5F_9, M-OCF_3; 181 (13) C_4F_7; 131 (51) C_3F_5; 100 (16) C_2F_4; 69 (100) CF_3.

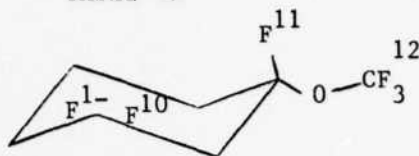CI(CH$_4$): 297 (1) $C_6F_{11}O$, M-F; 183 (35) $C_4F_7H_2$; 175 (48) C_5F_6H; 153 (23) C_5F_4O; 132 (100) C_3F_5H; 101 (29) C_2F_4H; 100 (13) C_2F_4; 69 (34) CF_3

[a]See ref. 11

[b]See ref. 10

SUPPLEMENTARY MATERIAL

TABLE 2a

F-Methoxycyclohexane

IR: 1300 s, 1270 vs, 1250 vs, 1230 s, 1195 vs, 1150 s, 1055 w, 995 s, 980 vs, 880 w, 850 m, 755 m, 730 m, 690 w, 660 w, 630 m, 610 m cm^{-1}

^{19}F nmr:[a]

$F_{12}(CF_3)$ $\phi = -53.83$ ppm, mult.
$F_{11}(-F)$ $\phi = -142.77$ ppm, mult.
F_1-F_{10}:   three overlapping AB quartets
$\phi = -122.79$ to -134.63 ppm
matching literature spectrum[a]

Mass Spectra

EI(70eV): 281 (4) C_6F_{11}, M-OCF_3; 259 (1) C_6F_9O; 231 (7) C_5F_9; 181 (7) C_4F_7; 131 (52) C_3F_5; 100 (6) C_2F_4; 69 (100) CF_3.

CI(CH$_4$): 161 (23) C_7F_4H; 131 (100) C_3F_5; 100 (17) C_2F_4; 69 (65) CF_3.

[a]See ref. 11

<div align="center">

SUPPLEMENTARY MATERIAL

TABLE 2b

</div>

F-Cyclopentanone

IR:[a,b] 1830 (vw), 1810 w, 1345 m, 1300 m, 1200 vs, 1145 w, 1115 w, 1010 m, 965 s, 725 m cm^{-1}

^{19}F nmr:[a]

ϕ = -126.52 ppm, m [4]
ϕ = -136.76 ppm, m [4]

Mass Spectra:[a]

EI(70eV): 228 (1) C_5F_8O, M; 200 (2) C_4F_8; 181 (2) C_4F_7; 131 (78) C_3F_5; 100 (100) C_2F_4; 69 (11) CF_3.

CI(CH_4): 209 (1), C_5F_7O, M-F; 182 (32) C_4F_7H; 132 (100) C_3F_5H; 101 (70) C_2F_4H.

[a]See ref. 8
[b]See ref. 6a

F-Cyclohexanone

IR [a,b] 1795 m, 1300 s, 1255 s, 1185 vs, 1220 m, 1080 m, 1025 w, 990 s, 960 vs, 805 w, 725 w, 615 w cm^{-1}

^{19}F nmr:[a] ϕ = -125.15 ppm, s [4]
ϕ = -133.23 ppm, m broad [6]

Mass Spectra[a]
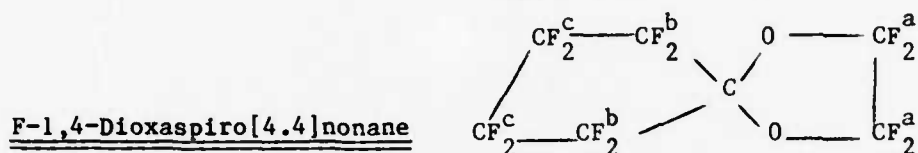
EI(70eV): 278 (2) $C_6F_{10}O$, M; 231 (1) C_5F_9; 181 (12) C_4F_7; 131 (100) C_3F_5; 100 (23) C_2F_4; 93 (5) C_3F_3; 81 (3) C_2F_3; 69 (10) CF_3.

[a]See ref. 8

[b]See ref. 6a

SUPPLEMENTARY MATERIAL

TABLE 2c

F-1,4-Dioxaspiro[4.4]nonane

IR:[a,b] 1405 w, 1310 s, 1275 s, 1245 s, 1210 vs, 1160 vs, 1080 m, 1060 m, 1035 m, 980 vs, 810 m, 725 m, 655 w, 595 m, 520 m cm^{-1}.

^{19}F nmr:[a]

ϕ = -85.32, s   [4] CF_{2a}

ϕ = -130.36 ppm, s   [4] CF_{2b}

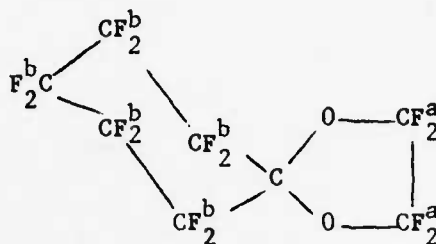ϕ = -131.69 ppm, s   [4] CF_{2c}

Mass Spectra:
   EI(70eV): 325 (4) $C_7F_{11}O_2$, M-F; 225 (5) $C_5F_7O_2$; 209 (6) C_5F_7O; 194 (48) $C_4F_6O_2$; 131 (100) C_3F_5; 100 (60) C_2F_4; 69 (19) CF_3.

   CI(CH$_4$): 325 (100) $C_7F_{11}O_2$, M-F; 117 (98) C_5F_3; 97 (19) C_2F_3O.

Elemental Analysis	%C	%F	%H
Calc. for $C_7F_{12}O_2$	24.44	66.26	0.00
Found	24.22	66.25	0.00

SUPPLEMENTARY MATERIAL

TABLE 2d

F-1,4-Dioxaspiro[4.5]decane

IR 1400 w, 1310 m, 1280 s, 1250 vs, 1220 s, 1190 vs, 1160 vs, 1050 w, 980 vs, 970 vs, 820 w, 730 w, 625 w, 600 w cm^{-1}.

^{19}F nmr: $\phi = -83.22$ ppm, s   [4]

         $\phi = -132.02$ ppm, m broad [10]

Mass Spectra[a]

EI(70eV): 375 (1) $C_8F_{13}O_2$, M-F; 259 (3) C_6F_9O; 244 (4) $C_5F_8O_2$; 181 (8) C_4F_7; 131 (100) C_3F_5; 100 (20) C_2F_4; 97 (13) C_2F_3O; 69 (31) CF_3.

CI(CH$_4$): 414 (11) $C_8F_{15}O_2H$, M+HF; 348 (4) $C_7F_{13}OH$; 232 (5) $C_4F_8O_2$; 182 (12) $C_3F_6O_2$; 172 (24) C_8F_4; 151 (18) C_3F_6H; 143 (13) C_4F_5; 131 (100) C_3F_5; 117 (66) C_5F_3; 100 (19) C_2F_4; 97 (71) C_2F_3O; 95 (42) C_5FO; 69 (1) CF_3.

Elemental Analysis	%C	%F	%H
Calc. for $C_8F_{14}O_2$:	24.38	67.49	0.00
Found	23.85	66.78	0.00

APPENDIX II

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